Formation of An Oxygen-Deficient Y_2O_3 Phase Under High Pressure

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Formation of An Oxygen-Deficient Y_2O_3 Phase Under High Pressure

Polycrystalline Y_2O_3 is the material of choice for IR applications since it has excellent optical properties in the visible, and near infra-red band.

Current processing methods yield polycrystalline Y_2O_3 with large grain size (> 100 μm), which limits the hardness and erosion resistance attainable.

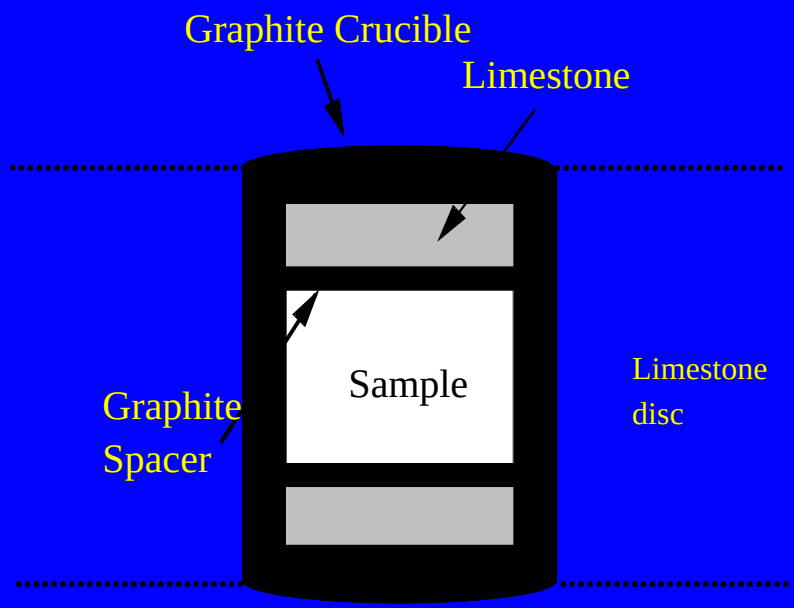
One way to improve strength is to develop an ultra-fine grained material with acceptable optical transmission properties.

It has been found recently that reversible transformation in consolidated Y_2O_3 under high pressure and temperature materials led to and extraordinary reduction in grain size .

Holding consolidated Y_2O_3 material under high pressure and temperature also yielded a novel surface structural modification

Phase Transformation under High pressure of consolidated Y_2O_3 samples

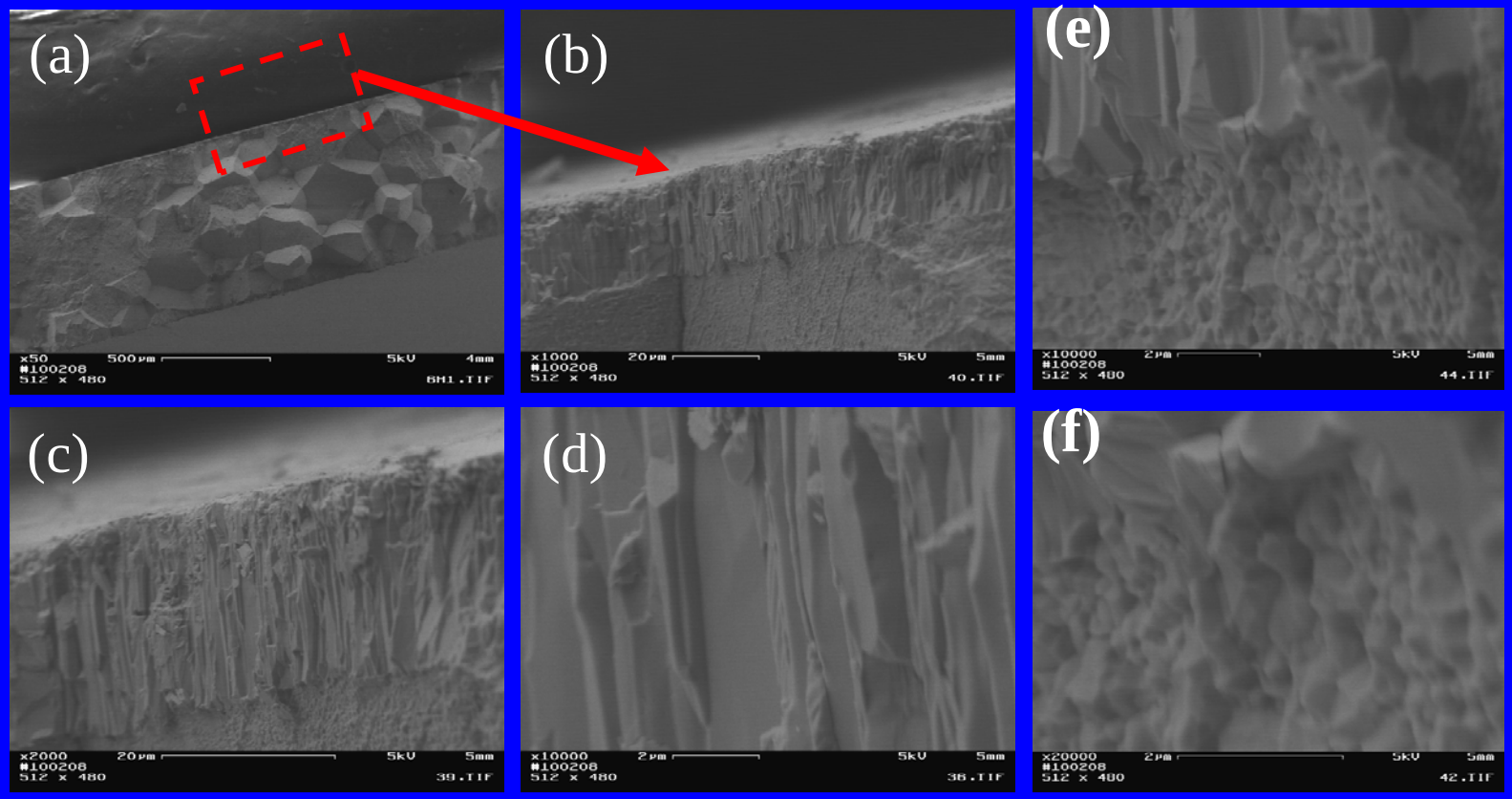
Pressure range 1- 10 GPa
Temperature 25-2000C



***Resistive heating* of graphite crucible enables rapid heating up to operating temperature**

- High pressure (8GPa/1000C/1-15min) has been utilized for grain size refinement (300 um → ~100 nm) via reversible transformation (cubic → monoclinic→ cubic)

Surface columnar grains and grains size refinement in Y_2O_3 after hot pressing at 8 GPa/1000ºC

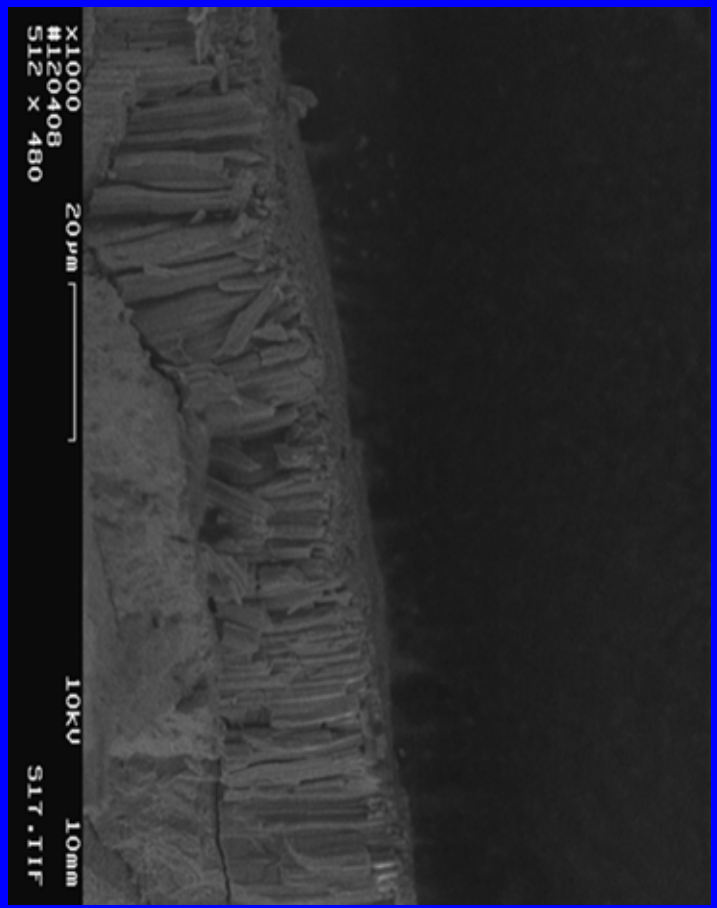


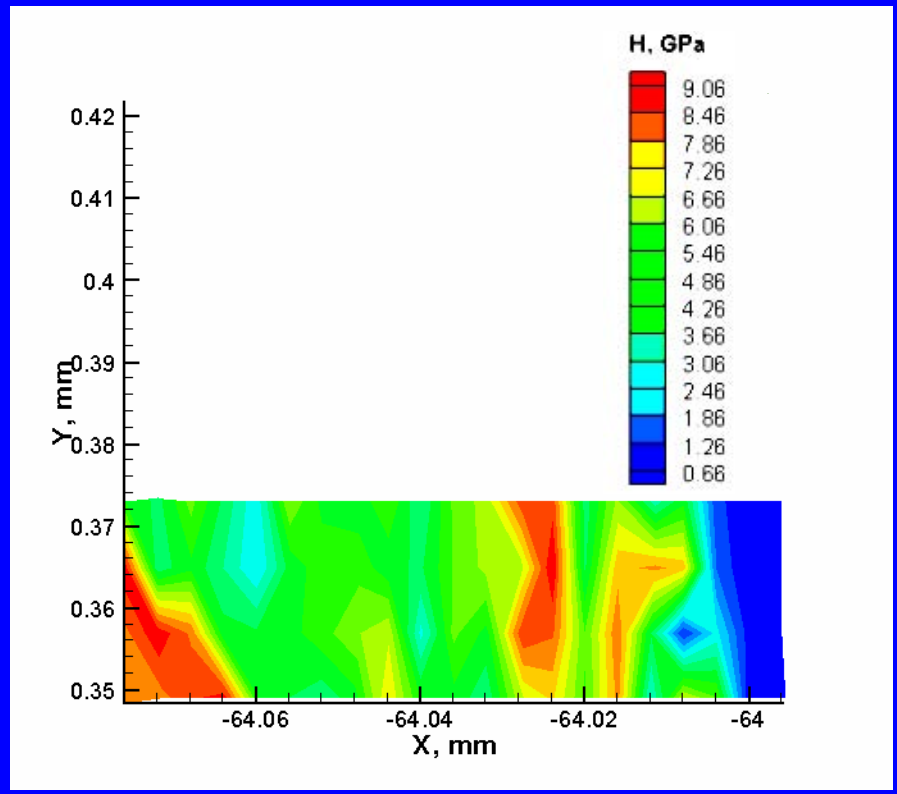
Sample was processed at 8 GPa/1000ºC for 6 hours

Predominantly intergranular fracture, apparently along grain boundaries of the *original* coarse-grained (~300 µm) cubic-Y_2O_3 has been observed

Sample's surface exhibit columnar grains while sample's interior was transformed under pressure into nano-grained (~100 nm) monoclinic-Y_2O_3

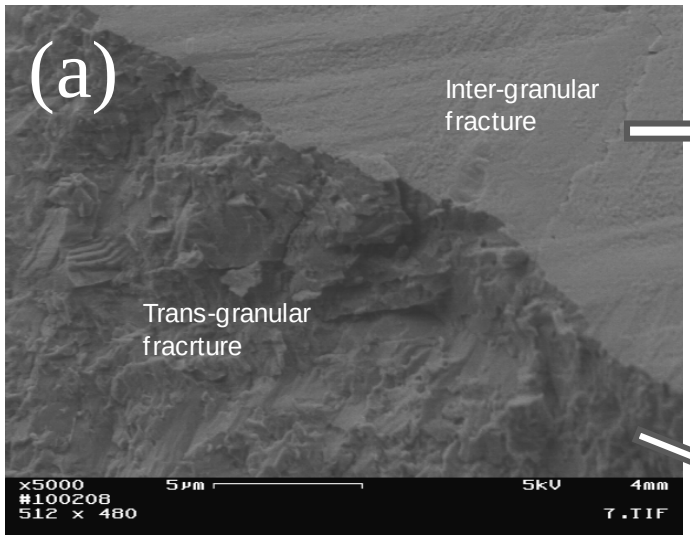
Hardness measurements using nano indentation

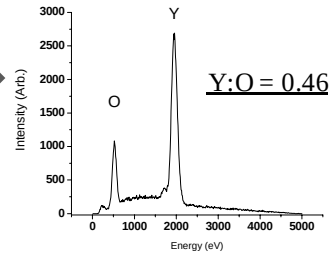


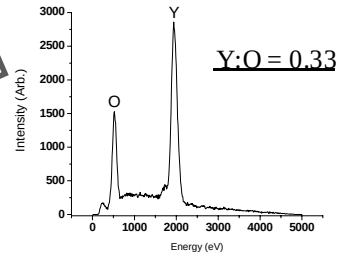


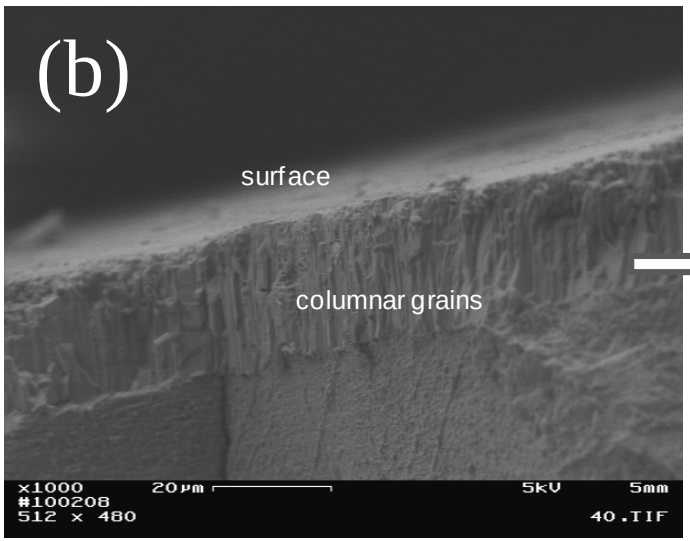
- **Columnar grains showed ~ an order of magnitude lower hardness than the sample interior**
- **This difference in hardness must be due to chemical and structural differences**

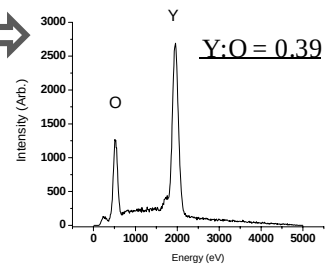
EDS analysis of fractured surface











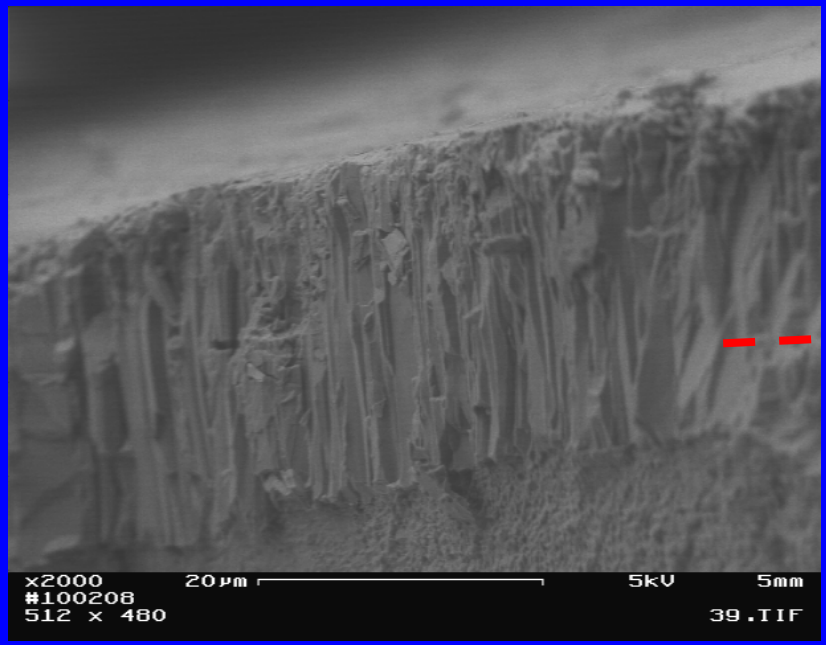
EDS analysis of a fractured sample, comparing the Y/O ratios for the smooth intergranular fracture, rough transgranular fracture, and faceted columnar-grained structure

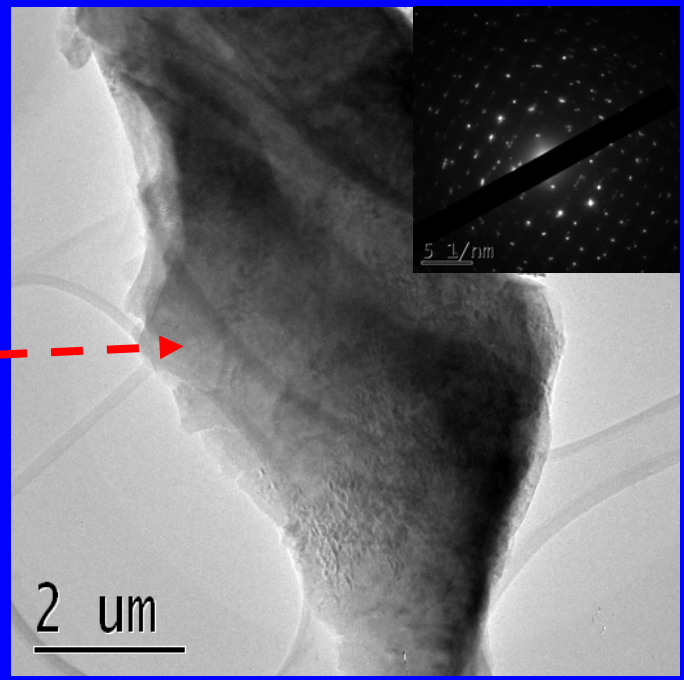
Y_2O_3 is reported to loose oxygen at high temperatures and a formation of $Y_2O_{2.88}$ is possible[1]

Y_2O_3 is reported to lose oxygen on heating at high temperatures; a stoichiometry of $Y_2O_{2.88}$ at 1800°C is reported.[1]

[1]. R. J. Ackermann, E. G. Rauh, and R. R. Walters, *J. Chem. Thermodyn., 2 [1] 139-149 (1970).*

TEM Analysis

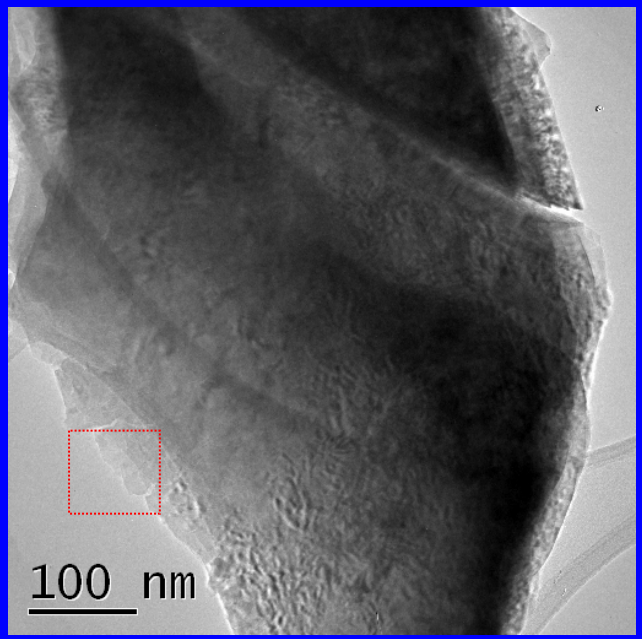




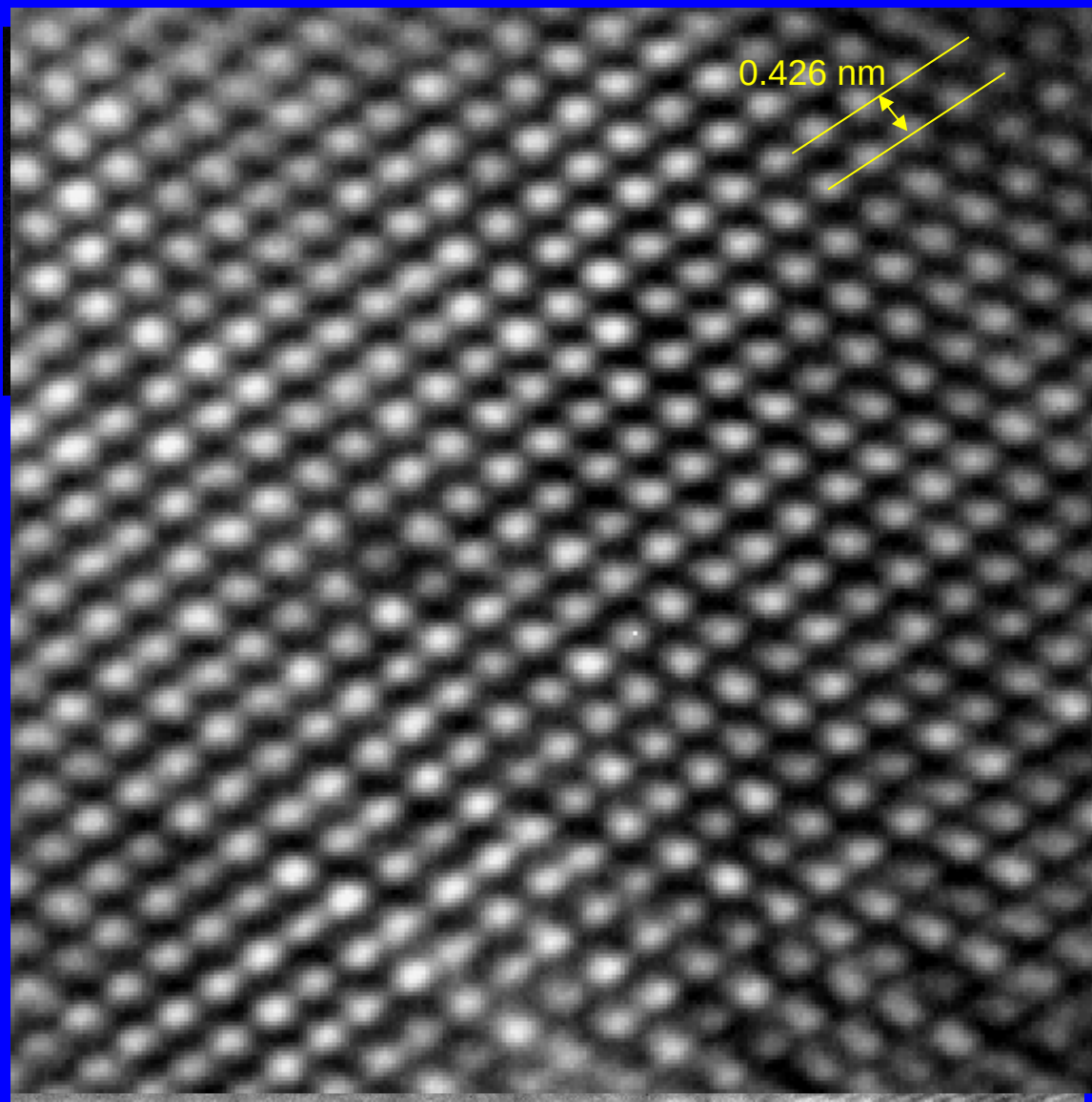
- **TEM sample has been prepared by scraping the surface onto copper grid**
- **Electron-transparent columnar grains with same dimensions and aspect ratio from SEM were observed**

Measured d-spacings (nm)	(hkl) Cubic, Ia-3
0.443	(211)
0.353	(220)
0.297	(222)
0.258	(400)
0.189	(440)

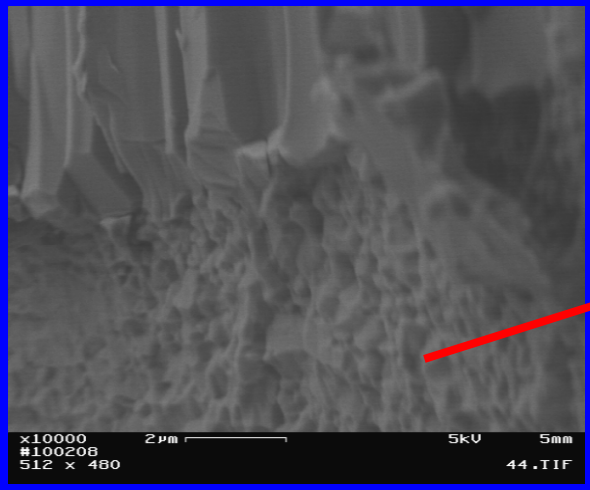
HRTEM imaging and FFT analysis

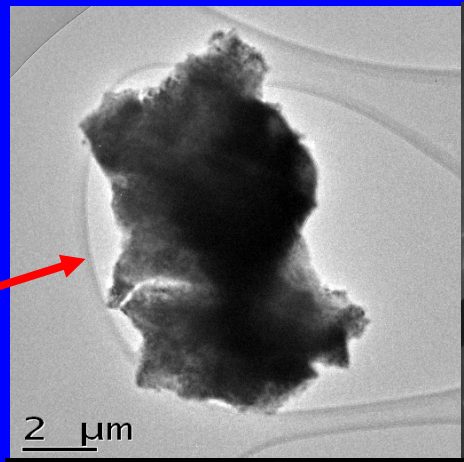


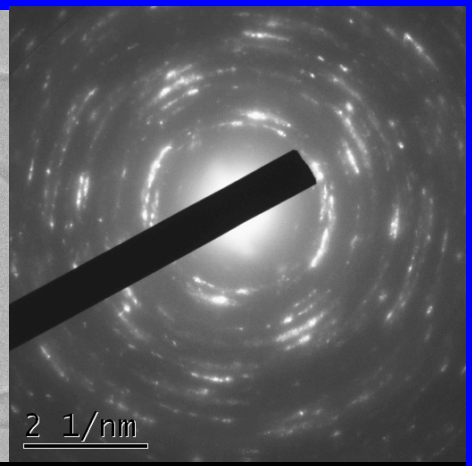
Spot#	d-spacing (nm)
1	0.426
2	0.233
3	0. 201
4	0.190

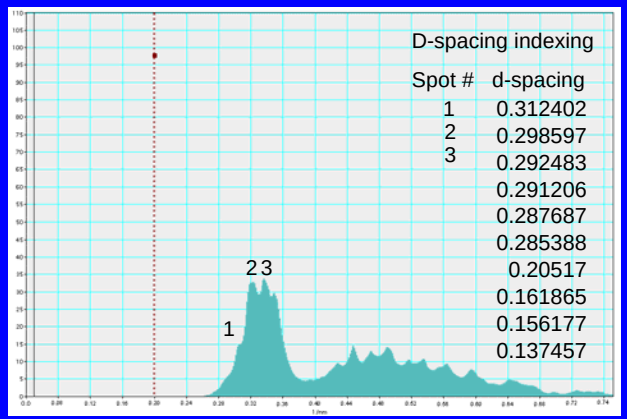


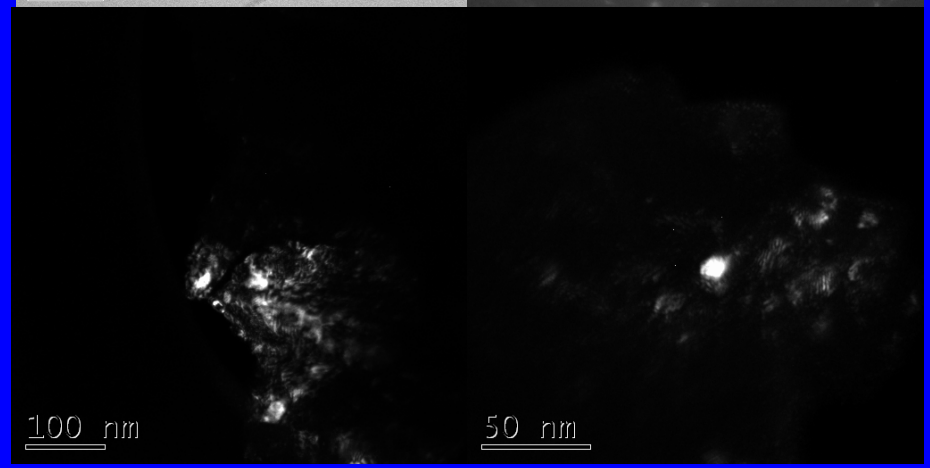
TEM Analysis-bulk











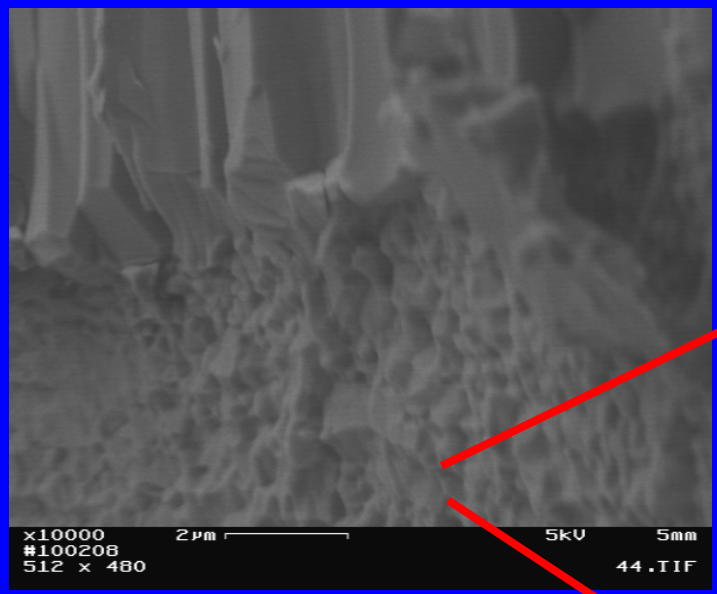
- TEM analysis confirms crystallite size < 50 nm
- Diffraction analysis confirms the presence of monoclinic phase

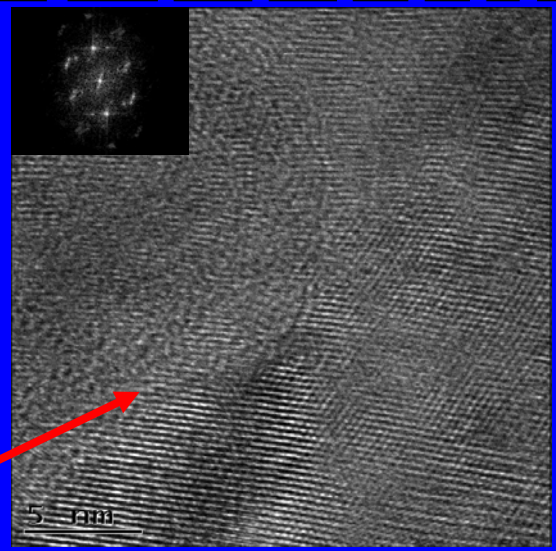
TEM Analysis-bulk

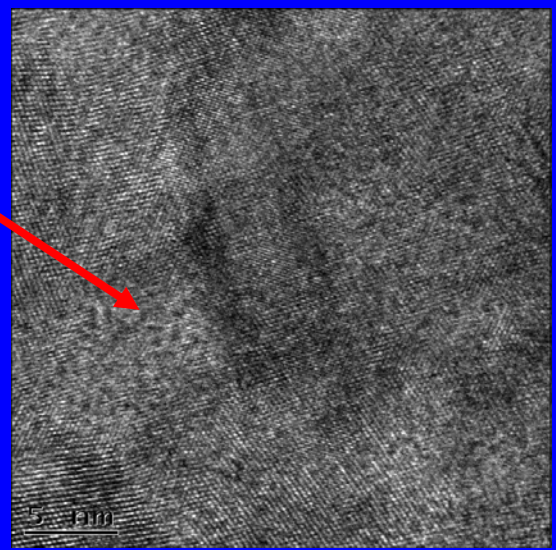
Monoclinic structure (c2/m)

Nano domains

Some defects (discontinuity in the lattice fringes)

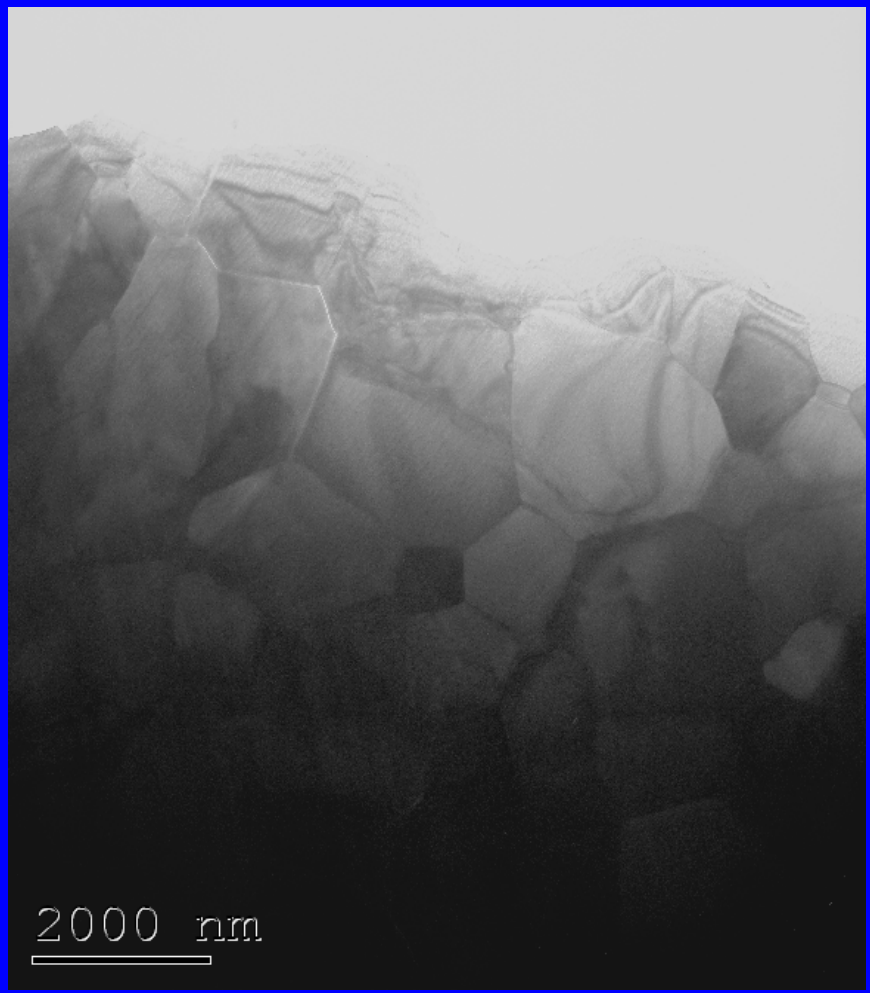




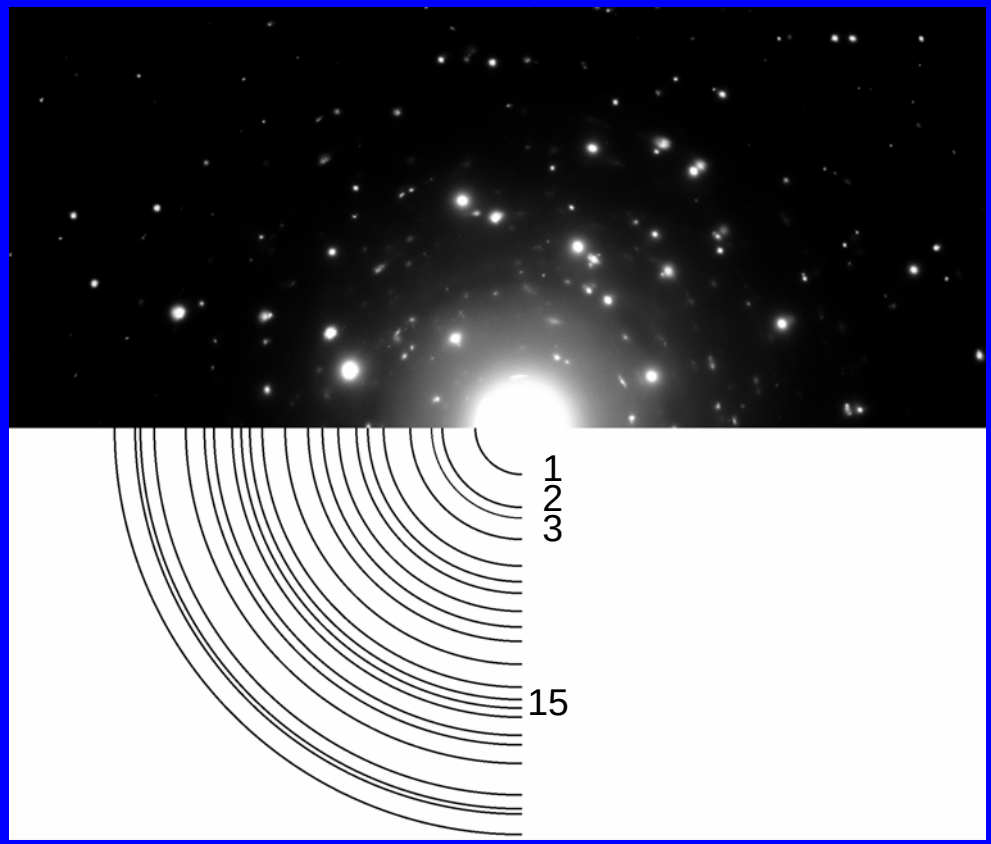


Measured d-spacings from FFT pattern (nm)	(hkl) Monoclinic,C2/m
0.319	(111)
0.292	(401)
0.289	(003)
0.267	(11-2)

columnar grains thinning via wedging process (tripod technique)

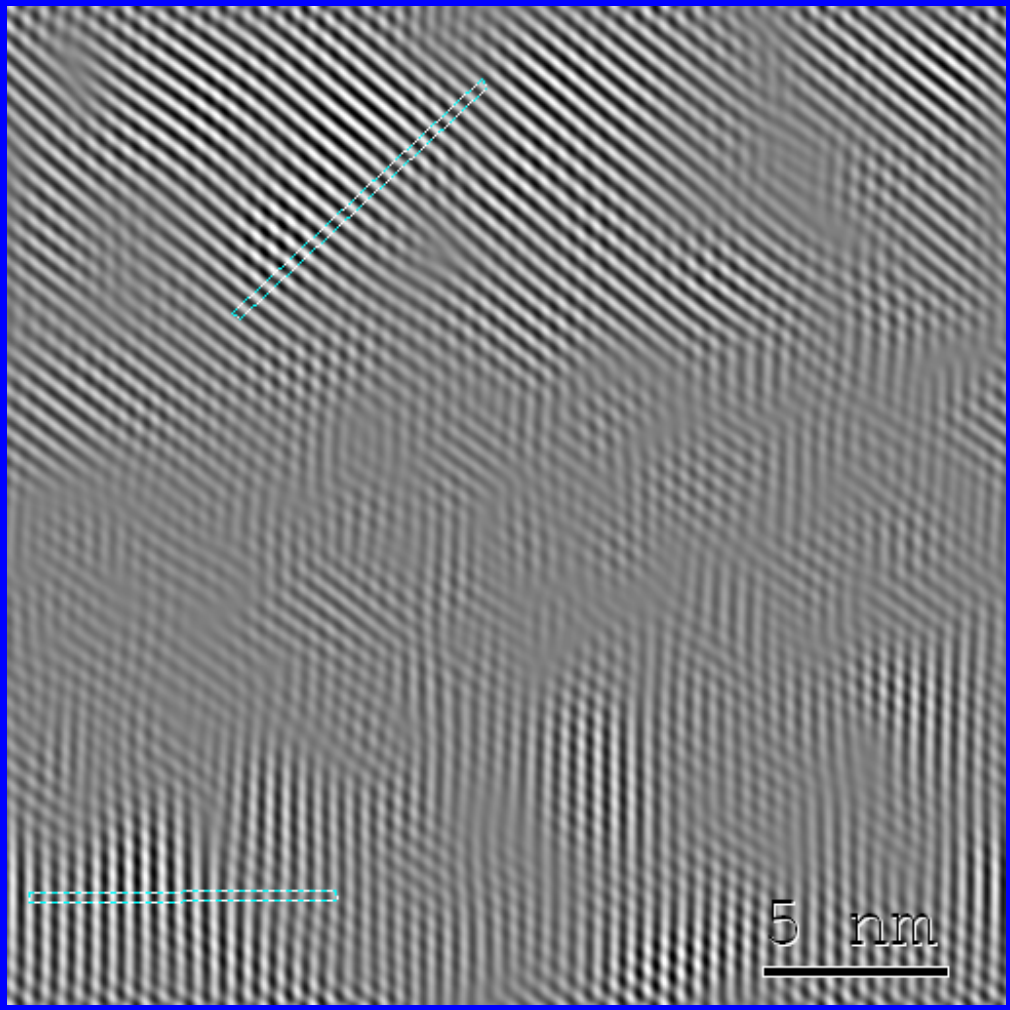


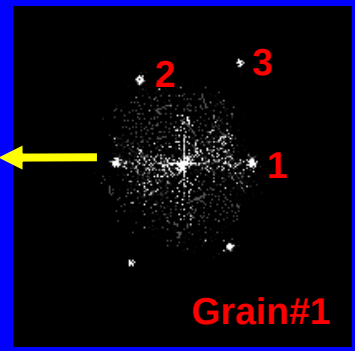
Selected Area electron diffraction analysis

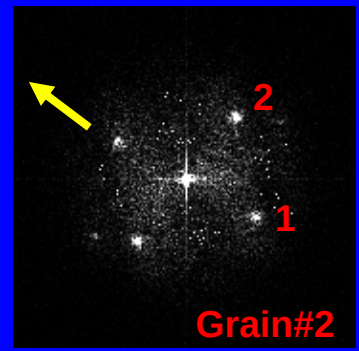


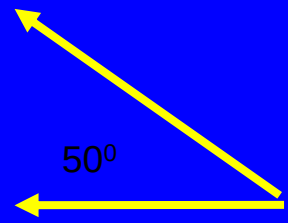
Spot#	d-spacing (nm)	(hkl)
1	0.431	211
2	0.381	220
3	0.308	222
4	0.248	411
5	0.223	332
6	0.208	134
7	0.187	440
8	0.172	611
9	0.161	145
10	0.145	046
11	0.132	800
12	0.126	653
13	0.122	831
14	0.119	752
15	0.112	664

HRTEM analysis of columnar grains



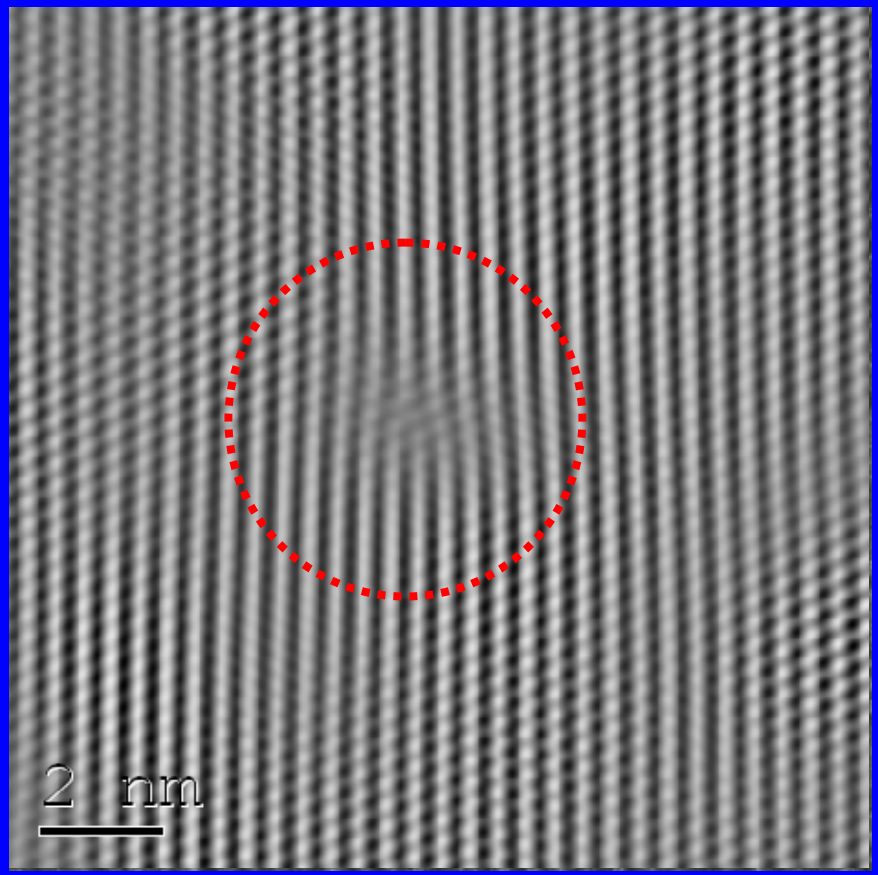


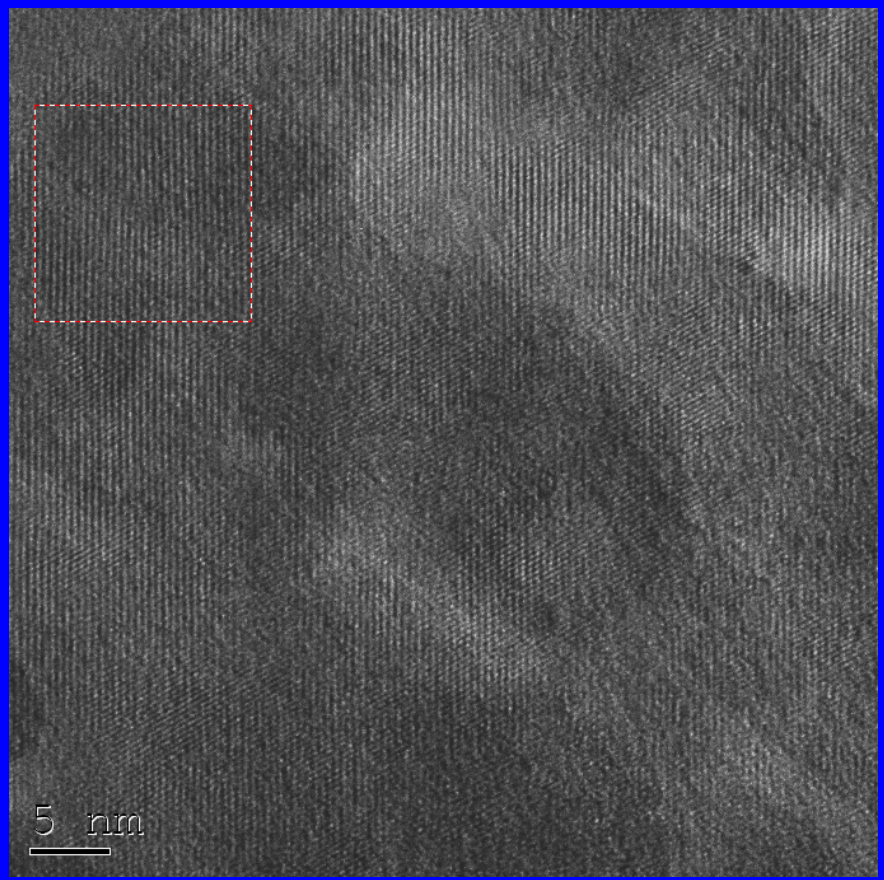




Spot #	d-spacing (nm)	Corresponding plane	
Grain#1			
1	0.429	211	ZA=0-11
2	0.307	222	
3	0.254	400	
Grain#2			
1	0.428	211	ZA=0-11
2	0.433	$21\bar{1}$	

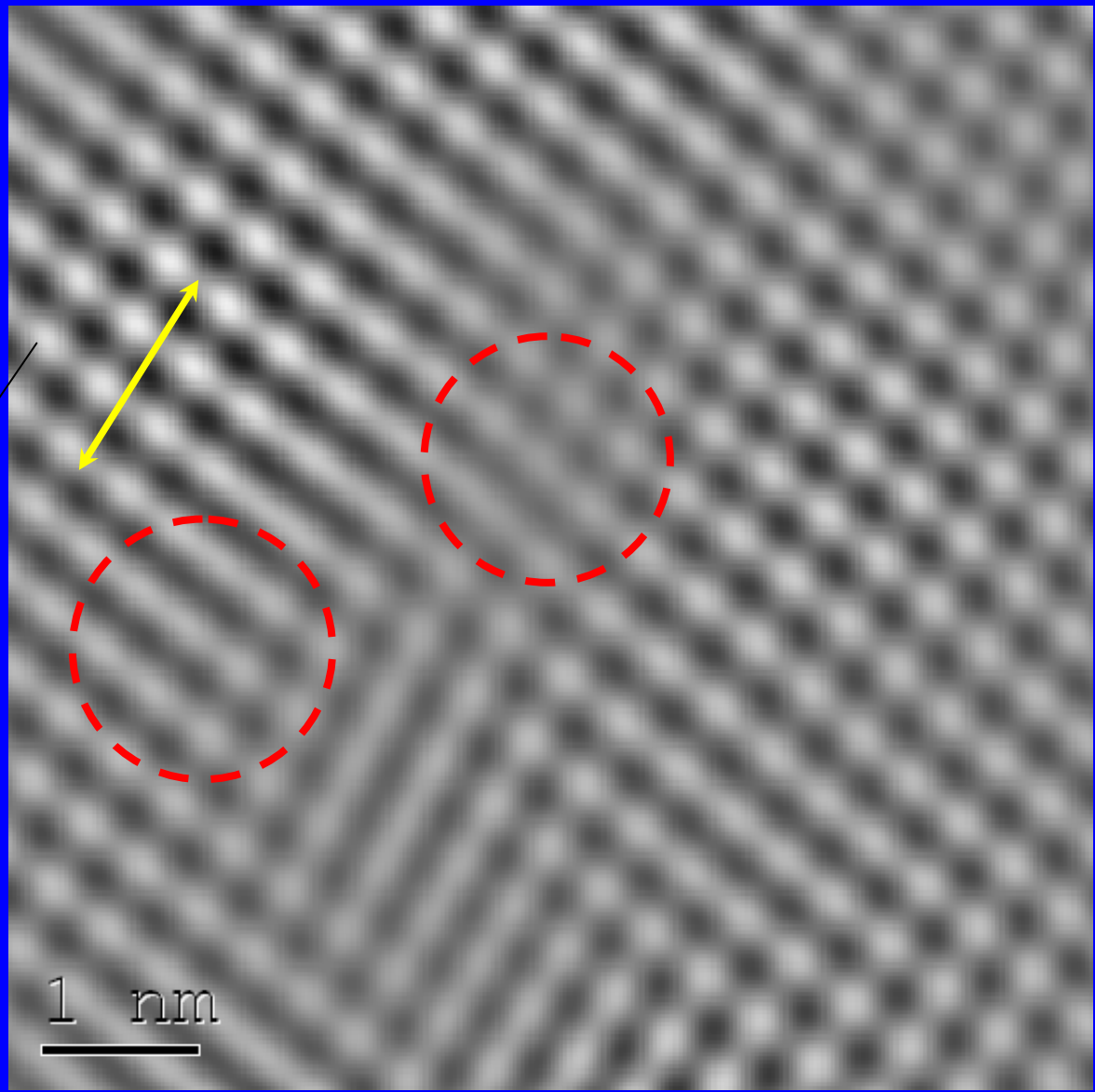
HRTEM analysis
Grain #1





Edge dislocation

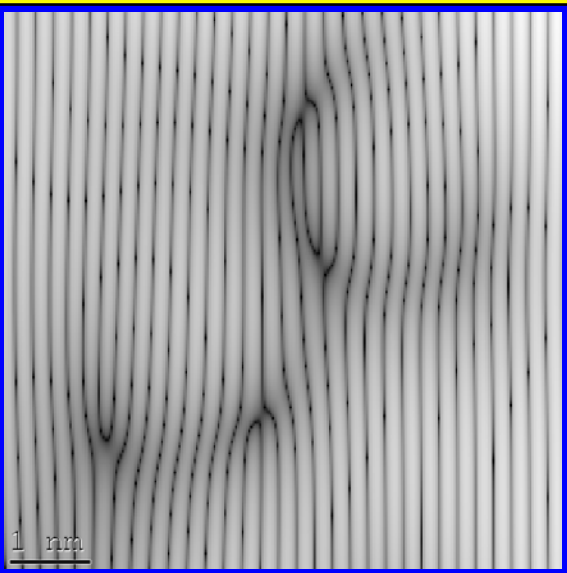
Interaction between two dislocations

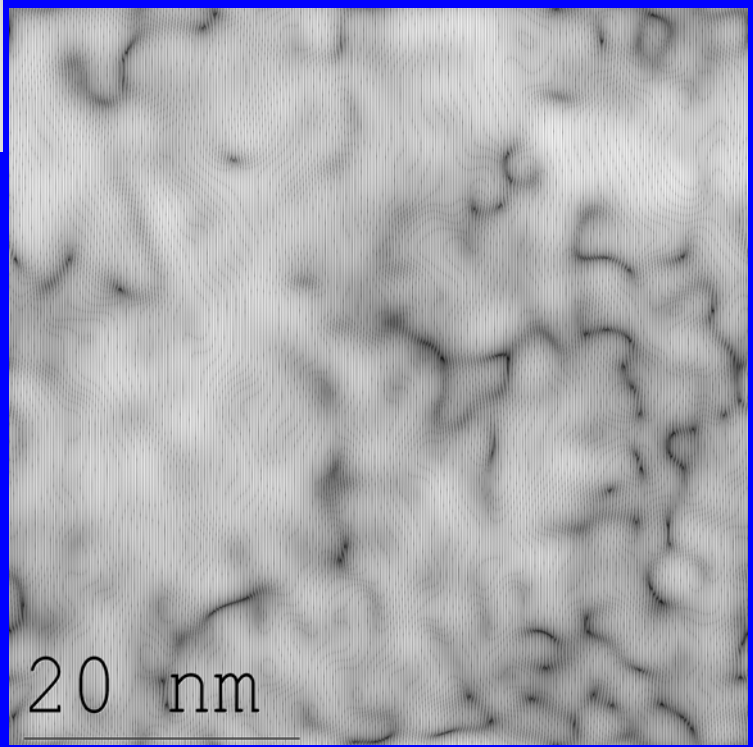


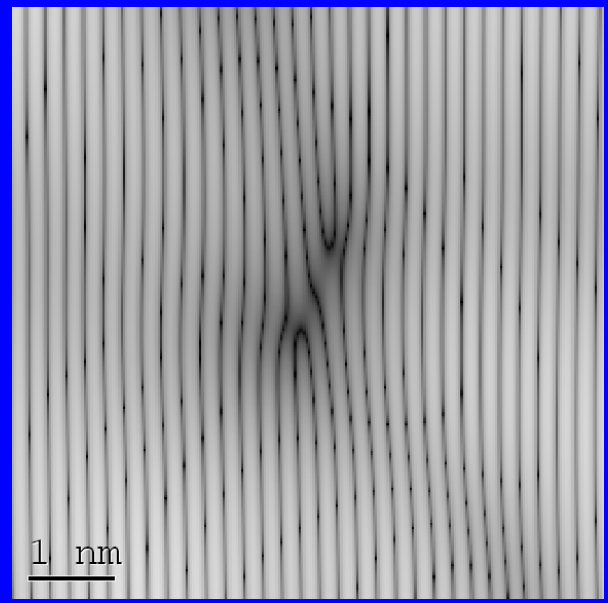
Burgers vector is
(a/2)[211]

An edge dislocation dipole (positive and negative pair) is separated by three lattice planes

Strain mapping from HRTEM







Summary-Conclusions-Future work

- A new oxygen-deficient columnar grains surface phase has been observed, due to reaction of the graphite crucible with the Y_2O_3 sample
- Such reductive decomposition is a promising technique for surface modification of ceramic materials
- Experiments to reverse the transformation are under way
- This technique will be investigated for other oxide phases

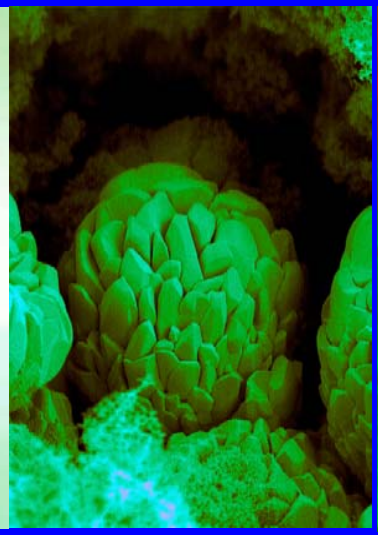
Acknowledgments

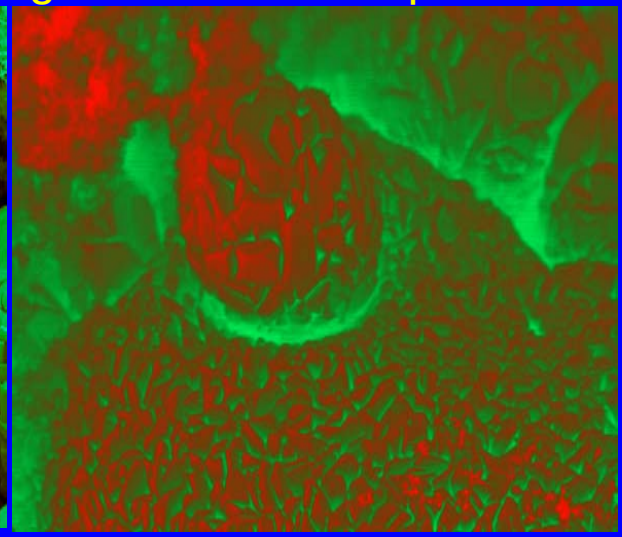
Support for this work from ONR and DARPA is gratefully acknowledged

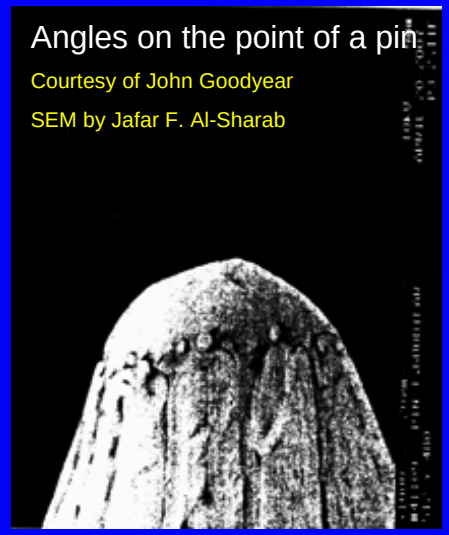
nano art work

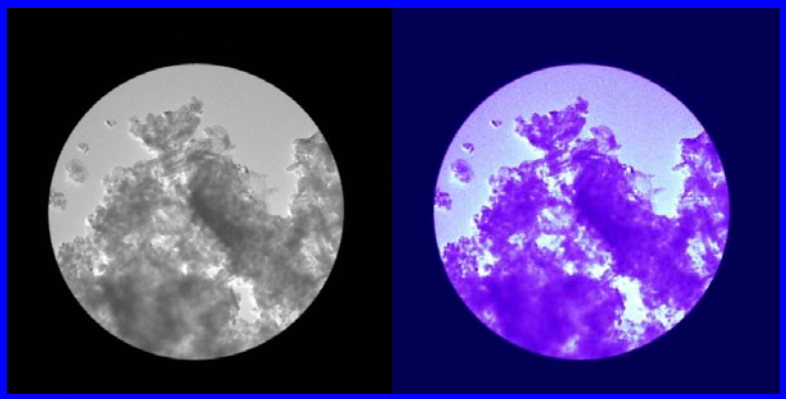
Artichoke

Artichoke-like tungsten oxide nano particles

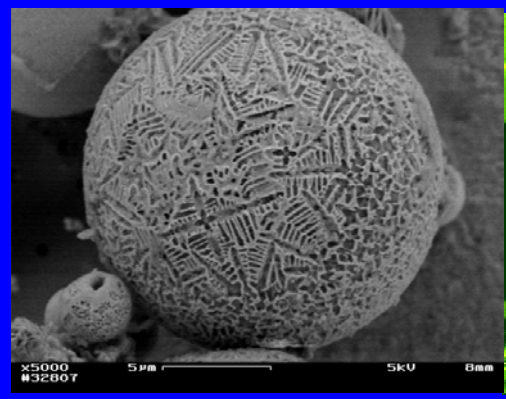


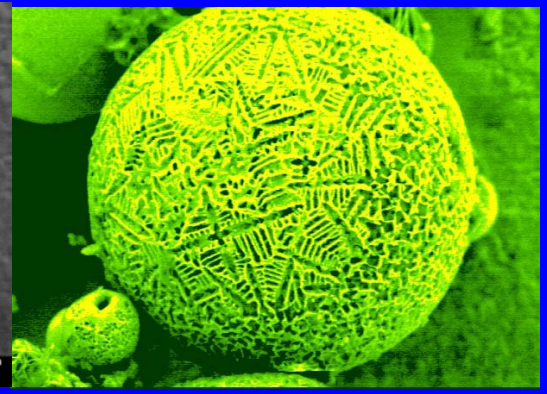






Magnesia-yttria nano particles with selected area aperture shadow





Plasma sprayed magnesia-yttria particles

thank you